

# Certificate

## Standard Reference Material® 2810

### Rockwell C Scale Hardness - Low Range

#### Serial Number:

This Standard Reference Material (SRM) is a transfer standard intended primarily for use in the calibration and verification of the performance of Rockwell hardness equipment using the Rockwell C hardness scale (HRC). Each SRM unit is a steel test block, nominally 64 mm in diameter and 15 mm thick, having a polished test surface described by a micro-engraved circle 52 mm in diameter as shown in Figure 1. Each SRM unit bears a unique serial number on the edge of the block.

SRM 2810 is individually certified by performing seven hardness measurements at specific locations on the test surface as indicated in Figure 1. Recommended test conditions, procedures, and precautions for the proper use of this SRM are provided in the Annex. In order to perform calibration or verification measurement over the full usable range of the HRC scale, this SRM may be used in conjunction with SRM 2811 Rockwell C Scale Hardness - Mid Range and SRM 2812 Rockwell C Scale Hardness - High Range.

Expiration of Certification: Certification of this SRM is valid within the uncertainties stated until 01 May 2006, provided the SRM is stored and handled in accordance with the instructions given in this certificate and its annex. Any mechanical damage or alteration to the surface, such as grinding or polishing, will invalidate the certification.

Maintenance of Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of certification, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Hardness measurements leading to the certification of this SRM were performed by S.R. Low and D.J. Pitchure with the assistance of R.E. Ricker of the NIST Metallurgy Division.

Statistical analysis was performed by W.S. Liggett of the NIST Statistical Engineering Division.

Geometric verification of the NIST Rockwell diamond indenter used in the certification of this SRM was conducted by J.F. Song and T.V. Vorburger of the NIST Precision Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by C.R. Beauchamp and N.M. Trahey.

Carol A. Handwerker, Chief Metallurgy Division

John Rumble, Jr., Acting Chief Standard Reference Materials Program

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Certified Hardness Values: This SRM is certified using two different certification modes: (1) the certified average hardness value over the entire test surface and (2) individually certified hardness values at specific untested locations on the test surface. The certified average hardness value (Certification Mode 1) provides the user with a comparison value for performing indirect verifications [1] of Rockwell hardness machines for the HRC scale. The individually certified values of hardness at specific untested locations (Certification Mode 2) provide a more complete characterization of the surface hardness of the block. The user can minimize the measurement uncertainty due to material non-uniformity by using Certification Mode 2.

Certification Mode 1: Certified Average Hardness Value: This value is an estimate of the average hardness across the test surface of the block. Hardness varies slightly across the surface due to material non-uniformity and can be modeled by a mathematical function. The average hardness value is defined as that hardness function integrated over the test block surface divided by the surface area.

The certified average hardness value is

 $\pm$  0.18 HRC

Uncertainty of the Certified Average Hardness Value: The expanded uncertainty, U, in the average certified hardness was calculated using  $U = ku_c$ , where  $u_c$  is the combined standard uncertainty calculated according to the ISO Guide [2], and k = 2 is the coverage factor. The value of  $u_c$  represents the combined effect of the individual sources of uncertainty listed in Table 1. These sources include uncertainties due to deviations of the NIST standardizing tester and indenter from the defined requirements for the Rockwell hardness test (forces, depth measurement, test cycle, and indenter shape) [1]. The value of  $u_c$  is calculated as follows

$$u_{\rm c} = \sqrt{{\sigma_1}^2 + {\sigma_2}^2 + {\sigma_3}^2 + {\sigma_4}^2}$$

Table 1. Sources of Uncertainty for the Certified Average Hardness Value

Туре		Uncertainty Source	Standard Uncertainty
$\sigma_1$	A	Material Uniformity and Measurement Repeatability	± 0.013 HRC
$\sigma_2$	A	Day-to-Day Reproducibility	± 0.056 HRC
$\sigma_3$	В	NIST Standardizing Hardness Tester	± 0.020 HRC
σ4	В	NIST Standardizing Indenter	± 0.067 HRC
$u_{c}$		Combined Standard Uncertainty	± 0.091 HRC
U		Expanded Uncertainty	± 0.18 HRC

The expanded uncertainty, U, indicates how well NIST's estimate of the true value of the average hardness of this SRM is. It should not be interpreted as the range in expected hardness values that would be measured across the test surface, nor is it a limit of acceptable hardness values for verifying Rockwell hardness equipment. Similarly,  $\sigma_1$ ,  $\sigma_2$ ,  $\sigma_3$ , and  $\sigma_4$  are uncertainty components that contribute to the overall uncertainty in the certified average hardness value and are not ranges of expected hardness results due to each source of uncertainty.

Certification Mode 2: Individually Certified Hardness Values for Specific Locations: This certification mode provides the user with individually certified hardness values at 11 untested locations on the SRM. The certified values at each location, defined in x-y coordinates and labeled with the letters A through K, are given in Table 2 and illustrated in Figure 2.

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Table 2. Individually Certified Hardness Values for Specific Test Block Locations

Location	x (mm)	y (mm)	Hardness (HRC)
A	0	23	± 0.21
В	20	12	± 0.21
C	20	- 12	± 0.21
D	0	- 23	± 0.21
E	- 20	- 12	± 0.21
F	- 20	12	± 0.21
G	0	12	± 0.20
H	10	6	± 0.20
I	10	- 6	± 0.20
J	- 10	- 6	± 0.20
K	- 10	6	± 0.20

The x-y coordinate system is such that location x = 0, y = 0 is at the block center (NIST indentation 4) and oriented with the NIST logo at the bottom of the block as illustrated in Figure 1.

Uncertainty of Individually Certified Hardness Values: The uncertainties in the individually certified hardness values for the locations reported in Table 2 are calculated using the same sources of uncertainty as for the certified average hardness values given in Table 1. A difference occurs in  $\sigma_1$ , the uncertainty component due to block uniformity and measurement repeatability. The remaining uncertainty components  $\sigma_2$ ,  $\sigma_3$ , and  $\sigma_4$  are the same for both types of hardness certification. These four uncertainty components are given in Table 3 at a one standard deviation level. These components are combined to calculate the standard uncertainty,  $u_c$ , to which the coverage factor k = 2 is applied to obtain the certified uncertainty U.

For the individually certified hardness values, the expanded uncertainty, U, indicates how well the best estimate of the true value of the hardness is at that specific location on the SRM. Similarly,  $\sigma_1$ ,  $\sigma_2$ ,  $\sigma_3$ , and  $\sigma_4$  are uncertainty components that contribute to the overall uncertainty in the certified hardness value at each specific location.

NIST Certification Procedure: Several test blocks were randomly selected from each production batch and covered with hardness indentations using the NIST standardizing hardness tester and diamond indenter. The NIST standardizing hardness tester is a specially designed machine using directly loaded dead-weights for applying the required forces, and a laser interferometry displacement sensor for measuring the depth of indentation. Hardness was measured by indentation tests in accordance with the Rockwell hardness principle [1] and following the test cycle given in the Annex.

The geometry of the NIST standardizing diamond indenter was verified using a stylus instrument [3]. The indenter measurements and expanded uncertainties are 199.06  $\mu$ m  $\pm$  1.97  $\mu$ m for the tip radius and 120.00 degree  $\pm$  0.02 degree for the cone angle.

By analyzing the hardness profile of this set of test blocks, information on material uniformity was developed. NIST determined that the hardness varies smoothly across the surface of each block and varies differently from block to block. This hardness variation is due primarily to the nature of steel and the manufacturing process.

Hardness measurements were then made at eight locations on the test surface of each SRM as illustrated in Figure 1. The first indentation, labeled "Seat", was made to ensure that the new test block was adequately seated on the anvil. "Seat indentation" data are potentially erroneous; thus, the resulting hardness value from this first indentation was discarded. The seven hardness tests that followed, labeled 1 through 7, were used in the determination of the SRM certified hardness values for that SRM.

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Table 3. Sources of Uncertainty for Hardness Values at Each of the Individual Locations Given in Table 2

	Uncertainty Source	Standard Uncertainty Locations A through F	Standard Uncertainty Locations G through K
$\sigma_1$	Material Uniformity & Repeatability	± 0.050 HRC	± 0.045 HRC
$\sigma_2$	Day to Day Reproducibility	± 0.056 HRC	± 0.056 HRC
$\sigma_3$	NIST Standardizing Hardness Machine	± 0.020 HRC	± 0.020 HRC
$\sigma_4$	NIST Standardizing Indenter	± 0.067 HRC	± 0.067 HRC
u <sub>c</sub>	Combined Standard Uncertainty	± 0.103 HRC	± 0.100 HRC
U	Expanded Uncertainty	± 0.21 HRC	± 0.20 HRC

Discussion: To illustrate the effect of the material non-uniformity, the test surface of the SRM block should be considered a collection of possible measurement locations. The metal at each location will have a unique hardness value that differs slightly from the hardness value at any other location. If it were possible to determine the hardness at every location, the collection of hardness values would be found to vary over a range, and an average hardness value for the entire test surface could then be calculated. The extent of this range defines the non-uniformity in hardness for the test surface. The smaller the range in hardness values, the more uniform is the block. Thus, for this SRM, the hardness at individual locations will vary over a range that extends both above and below the average hardness value of the test surface.

As a consequence of this hardness non-uniformity, a single certified hardness value does not completely describe the hardness of the test surface. Thus, two types of hardness certification are provided in this certificate: (1) the certified average hardness and (2) individually certified hardness values at untested locations. The certifications are based on the seven NIST hardness measurements and on modeling hardness as a smooth random function across the test surface. These two types of certifications define the hardness of the test block in distinctly different ways. The user must keep this in mind when using these certified values and the associated uncertainties.

In general, the closer two measurements are made, the closer the hardness values will be to each other, limited by minimum spacing considerations. The method used to predict hardness values at untested locations is based on a geostatistics formula [4] that models the hardness across the surface of a block as a smooth random function described by a semivariogram. The semivariogram is a mathematical model that describes how the measured hardness difference between any two test block locations relates to the physical spacing that separates the two locations. In statistical terms, this semivariogram gives one half the variance of the hardness difference between any two locations on the test block. Thus, the square root of twice the semivariogram is the standard deviation of this difference.

For this SRM, the semivariogram is given by a simple function of Euclidean distance. Consider two points,  $s_i$  and  $s_j$ , separated by the distance, d (in mm), where

$$d = \sqrt{(x_i - x_j)^2 + (y_i - y_j)^2}$$

The semivariogram is given by

$$\gamma (s_i - s_j) = \begin{cases} 0 & \text{if } d = 0 \\ c_o + c_e \left[ 1 - \exp(-d/a_e) \right] & \text{if } d \neq 0 \end{cases}$$

where  $c_o$ ,  $c_e$ , and  $1/a_e$  are given in Table 4. In addition to the semivariogram, the calculation of certified hardness values required the seven hardness readings obtained by NIST, given in Table 5.

Table 4. Semivariogram Coefficients that Describe Uniformity and Repeatability

Coefficients	Values
c <sub>o</sub>	0.0008
C <sub>e</sub>	0.9770
1/ a <sub>e</sub>	0.0001

Table 5. NIST Hardness Measurements for Individual Test Block Locations

Location	X	у	Hardness Value (HRC)
1	- 10	17	
2	20	0	
3	- 10	- 17	
4	0	0	
5	10	17	
6	10	- 17	
7	- 20	0	

The certified average hardness value is based on the seven NIST hardness measurements and on the hardness prediction function discussed above. This average value is defined as the hardness function integrated over the test block surface divided by the surface area, and as such, is the average of the predicted hardness values for *all* test surface locations, and *not* the arithmetical average of the seven NIST measurements. However, since the locations chosen for the seven NIST measurements provide a good representation of the surface, the certified average hardness is close in value to the arithmetical average. The NIST certified average hardness value is only somewhat analogous to the certified value assigned to a commercially calibrated hardness test block that *is* typically calculated as the arithmetical average of several calibration measurements.

This certificate also provides individually certified hardness values for 11 specific untested locations on the test surface of the SRM, given in Table 2. These certified hardness values and their associated uncertainties were calculated using a mathematical formula [4], which includes the semivariogram given above and the three coefficients given in Table 4. All that is required to perform these calculations are the values and locations for the seven NIST measurements given in Table 5, and the x-y coordinates of the locations for which hardness values are to be determined. Using this same formula, additional certified hardness values at other untested locations may be calculated by the user [5,6]. The formulas may also be used for calculating various averages of the hardness at two or more arbitrary locations.

Traceability: This SRM can be used to demonstrate that a user's measurements are traceable to the HRC scale as determined by NIST; however, the procedures required to demonstrate traceability are varied depending on the user's needs. In general, acceptable transfer of the NIST hardness values to the user can be accomplished in three ways: (1) verification within tolerances - verifying that the user's measurements are in agreement with NIST certified values within acceptable tolerance limits; (2) verification within uncertainties - verifying that the user's measurements are in agreement with NIST certified values within the uncertainty ranges of the user's and NIST's measurements; and (3) calibration - calibration of the performance of the user's hardness machine to the NIST certified values by making appropriate mathematical corrections, and then verifying that the user's corrected values are in agreement with NIST certified values within the uncertainties of the user's and NIST's measurements. Each of the three methods requires differing levels of effort by the user to determine the accuracy in their measurement.

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Instructions for Use: This SRM is intended for use with all equipment capable of conducting an HRC indentation hardness test. The HRC hardness test is defined as requiring the use of a Rockwell spheroconical diamond indenter and the following test forces: a preliminary force of 98 N (10 kgf); an additional test force of 1373 N (140 kgf) that, when combined with the preliminary force, generates the total test force of 1471 N (150 kgf). The Rockwell hardness tester should be set up and operated in accordance with the recommendations of the manufacturer and as required by applicable test method standards. All hardness measurements must be made within the circle engraved on the test surface of the SRM. The Annex provides recommended test conditions, procedures, and precautions for the proper use for this SRM.

**Storage and Handling:** This SRM is made of steel, and thus is susceptible to scratches, dents, and other mechanical damage, particularly on the top and bottom surfaces, as well as being susceptible to corrosion from moisture and reaction to skin oils. The SRM is wrapped in corrosion resistant paper and packaged in a protective wooden box.

When not in use, the SRM should be rewrapped and stored in the box or in a manner having equivalent or better mechanical and corrosion protection.

The user should wear protective gloves, such as lint-free cotton or powder-free latex gloves while handling the SRM. If gloves are not worn, the SRM should be cleaned with ethyl alcohol or other suitable cleaning agent, and dried thoroughly before storing. The SRM should be held only by the circular edge to avoid excessive contact with the top and bottom surfaces.

The SRM storage area should be maintained at 23 °C  $\pm$  2 °C (73 °F  $\pm$  5 °F) with a relative humidity of 50 % or less.

#### REFERENCES

- [1] ASTM E 18, Standard Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials, ASTM, Philadelphia, PA (latest published version).
- [2] Guide to the Expression of Uncertainty in Measurement, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993); see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC, (1994); available at <a href="http://physics.nist.gov/Pubs/">http://physics.nist.gov/Pubs/</a>.
- [3] Song, J.F., Rudder, F.F. Jr., Vorburger, T.V., and Smith, J.H., "Microform Calibration Uncertainties of Rockwell Diamond Indenters," Journal of Research of the National Institute of Standards and Technology, 100, Nov.-Dec., (1995).
- [4] Cressie, N.A.C., Statistics for Spatial Data, John Wiley & Sons, Inc., (1991).
- [5] Liggett, W.S., Low, S.R., Pitchure, D.J., and Song, J., "Capability in Rockwell C Scale Hardness," Journal of Research of the National Institute of Standards and Technology, 105, Jul.-Aug., (2000).
- [6] Low, S.R., "NIST Recommended Practice Guide, Rockwell Hardness Measurement of Metallic Materials," NIST Special Publication 960-5, (2001).

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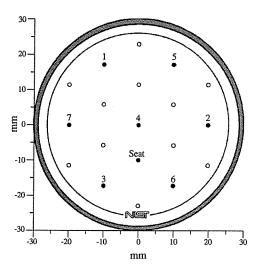


Figure 1. Test surface of SRM indicating the location and sequence of certification indentations (shown as black dots), and illustrating the appearance of the engraved circle and NIST logo. The labels 1 through 7 and "Seat" shown on the figure indicate the indentation sequence used in certification.

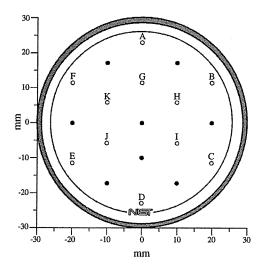


Figure 2. Test surface of SRM illustrating the locations (shown as open circles labeled A through K) of individually certified hardness values given in Table 2.

Certificate Revision History: 16 October 2001 (This revision reflects changes in the uncertainty values Tables 1, 2, 3, and the semivariogram coefficients in Table 4, in addition to editorial changes); 18 March 1998 (original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet <a href="http://www.nist.gov/srm">http://www.nist.gov/srm</a>.

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# Annex

## Standard Reference Material® 2810

### Rockwell C Scale Hardness - Low Range

#### Recommendations for Use

**Test Environment:** It is recommended that the hardness tester to be calibrated or verified be kept in a temperature and humidity controlled environment maintained at 23 °C  $\pm$  2 °C (73 °F  $\pm$  5 °F) and at a relative humidity of 50 % or less. The tester must also be in a location that is free from shock or vibration that could affect the hardness measurements.

**Anvil:** Use of a flat anvil is recommended (i.e., an anvil that can support the SRM for indentation anywhere on its test surface). However, when calibrating or verifying a hardness tester, the same anvil must be used with the SRM that will be used on the tester after the calibration or verification.

Cleaning the Anvil and Indenter: The tester anvil and indenter diamond tip should be thoroughly cleaned per recommendations from the manufacturer. In the absence of cleaning instructions from the manufacturer, it is recommended that the anvil and indenter be cleaned with ethyl alcohol, and dried using a lint free paper towel or cloth. Then, the surfaces should be blown clean of dust using filtered air, such as from a commercial compressed air can or bottle. **DO NOT** blow clean by mouth.

Cleaning the SRM: Prior to use, it is recommended that the SRM be cleaned. A recommended method for cleaning the SRM is to gently wipe the top and bottom surfaces with clean cotton thoroughly wetted with ethyl alcohol. The metal surfaces should immediately be dried using a soft lint-free paper towel or cloth before the alcohol evaporates. This cleaning must be performed in a manner that prevents a residue from remaining on the top or bottom surfaces. The alcohol cleaning should be followed by blowing the surfaces clean of dust with filtered air. The top and bottom surfaces should NOT be touched after cleaning.

Seating the Anvil and Indenter: Prior to making hardness indentations on the SRM, the tester anvil and indenter must be adequately seated. This can be accomplished by performing standard hardness tests on a material having a hardness value equal to or higher than the stated value of the SRM. The seating tests should be repeated until successive measurement values show no trend of increasing or decreasing hardness.

Placement of the SRM on the Anvil: Immediately before placing the SRM on the tester anvil, the top surface of the anvil and the bottom surface of the SRM should be blown free of dust as before. The SRM should be placed at once on the tester anvil before dust can return. The top test surface of the SRM should be blown free of dust prior to testing and occasionally during the period of use. When a flat anvil is used, the SRM should be slid several times back and forth over the surface of the anvil to help seat the block on the anvil. Any time the SRM is lifted from and replaced on the anvil, the procedure described above in this paragraph should be repeated. When a spot anvil is used, i.e., an anvil having a much smaller diameter than that of the SRM block requiring the block to be additionally supported at the start of the test when testing at locations other than at the center, extreme care should be practiced to ensure that the test block is supported parallel to the anvil until the indenter contacts the block and begins application of the preliminary force of 98 N (10 kgf).

Carol A. Handwerker, Chief Metallurgy Division

Gaithersburg, MD 20899 Annex Issue Date: 16 October 2001

John Rumble, Jr., Acting Chief Standard Reference Materials Program **Preliminary Indentation:** When a flat anvil is used, at least one preliminary HRC test should be performed at any location on the test surface of the SRM. The preliminary test will help seat the SRM on the anvil. The measured hardness value of the preliminary test should be ignored. A preliminary indentation is not necessary when using a spot anvil.

**Indentation Location:** The user is cautioned not to make any indentation such that it contacts a previous indentation, the engraved circle, or the NIST logo. No indentation should be made outside the engraved circle; doing so may damage the indenter. Under no circumstance shall an indentation be made on the bottom surface of the block.

**Test Cycle:** This SRM unit has been certified by performing Rockwell C scale hardness tests using a specific test cycle. The HRC test cycle may be characterized by specifying four test cycle parameters that have been determined to have a significant influence on the measurement results. The four parameters are: (1) preliminary test force dwell time; (2) total force dwell time; (3) recovery dwell time; and (4) indenter velocity during application of the additional force or additional force application time. These parameters are illustrated in Figure A1 and defined as follows:

**Preliminary Test Force Dwell Time** - the time period beginning when the preliminary force is fully applied and ending when the first baseline measurement of indenter depth is taken.

Total Force Dwell Time - the time period during which the total force is fully applied.

**Recovery Dwell Time** - the time period beginning when the additional test force is fully removed returning the applied force to the preliminary force level and ending when the final measurement of indenter depth is taken.

Additional Force Application Time - the time period during the application of the additional force. This parameter may also be specified in terms of the velocity of the indenter during this time period.

The test cycle parameters used for the certification of this SRM are given in Table A1.

Parameter	Value	NIST Tolerance
Preliminary Test Force Dwell Time	3 s	± 0.1 s
Total Force Dwell Time	5 s	± 0.1 s
Elastic Recovery Dwell Time	4 s	± 0.1 s
Indenter Velocity	40 μm/s constant	± 2 μm/s

Table A1. Specifications for the SRM Test Cycle Parameters

To minimize the uncertainty in the hardness measurement, a test cycle should be used that replicates, as closely as possible, the SRM certification test cycle parameters listed in Table A1. Deviations from the SRM test cycle in dwell times or force application rate will result in measured hardness values that deviate from measurements made using the SRM certification test cycle.

**Indentation Spacing:** Rockwell hardness measurements may be influenced by nearby previously made indentations. For this SRM, a hardness measurement will not be measurably affected by an adjacent Rockwell C scale indentation that is located a distance of 7 mm or greater, center to center spacing, from the measurement location. It is recommended that no indentation should be made closer than 5 mm, center to center spacing, from a previous indentation. For hardness measurements within 5 mm to 7 mm distance of only one single previous indentation, the resulting HRC value may be increased as much as 0.05 HRC. Measurements made within 5 mm to 7 mm distance of *multiple* previously made indentations will produce an even higher elevation in HRC. The user should consider this possible elevation of hardness when making measurements on this SRM. Table A2 lists the effect of indentation-to-indentation spacing for this SRM. In addition to this indentation-to-indentation spacing restriction, no HRC measurement should be made within 1 mm of the engraved circle or the NIST logo.

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Table A2. Effect of Indent Spacing

Separation Distance from One Indentation (Indentation center to center)	Effect on Hardness	
7 mm or greater	No interaction	
5 mm to 7 mm	Increase in hardness of up to 0.05 HRC	
less than 5 mm	Not recommended	

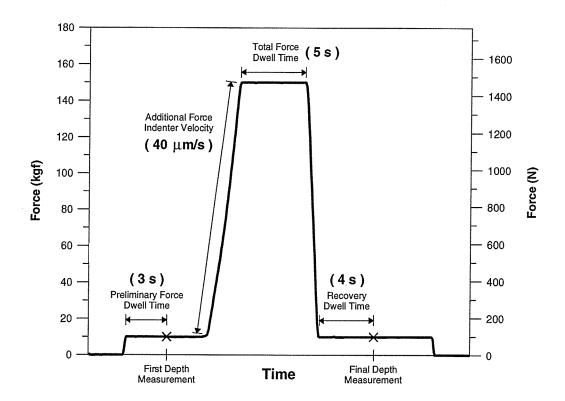


Figure A1. Illustration of the parameters of the HRC Test Cycle Used in the Certification of the SRM

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